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13. ABSTRACT (Maximum 200 words)

A series of organoindium-phosphides including $(Me_3CCH_2)_2InPRR'$ (R = R' = Et, C_6H_{11}; R = C_6H_{11}, R' = H; R = Me, R' = Ph) and $(Me_3SiCH_2)_2InP(Me)Ph$ have been prepared and characterized. The characterization data include partial elemental analyses (C, H), ^1H and ^{31}P NMR and IR spectral studies, physical properties, cryoscopic molecular weight studies (with the exception of $(Me_3CCH_2)_2InP(C_6H_{11})_2$), and X-ray structural studies of $[(Me_3CCH_2)_2InPEt_2]_2$ and $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$. The compound $(Me_3CCH_2)_2InPEt_2$ is observed to be a dimer in benzene solution; $(Me_3CCH_2)_2InP(H)(C_6H_{11})$ exists as a dimer-trimer equilibrium mixture whereas $(Me_3CCH_2)_2InP(Me)(Ph)$ and $(Me_3SiCH_2)_2InP(Me)(Ph)$ are trimers. NMR studies are consistent with $(Me_3CCH_2)_2InP(C_6H_{11})_2$ being a dimer in benzene solution but the

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concentration was too low to confirm the conclusion by cryoscopic molecular weight studies. The compound $[(Me_3CCH_2)_2InPEt_2]_2$ crystallizes in the monoclinic space group C2/c with a = 12.972(2)Å, b = 15.539(5)Å, c = 18.255(3)Å, β = 99.84(1)°, V = 3625.6 Å^3 and Z = 4 (dimeric molecules). The molecule contains a planar In_2P_2 core with In-P distances of 2.623(2) and 2.641(2)Å. The compound $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$ crystallizes in the triclinic space group $P\bar{1}$ with a = 11.272(3)Å, b = 13.992(5)Å, c = 20.539(9)Å, α = 95.37(5)°, β = 104.83(4)°, γ = 109.14(4)°, V = 2901.9 Å^3 and Z = 2 (trimeric molecules). The molecule has an In_3P_3 six-membered ring which is in the twist-boat conformation with In-P distances ranging from 2.613(3) to 2.659(2)Å.

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Synthesis and Characterization of a Series of Organoindium-Phosphides Including Molecular Structures of $[(Me_3CCH_2)_2InPEt_2]_2$ and $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$

by

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Synthesis and Characterization of a Series of Organoindium-Phosphides Including Molecular Structures of $[(Me_3CCH_2)_2InPEt_2]_2$ and $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$.

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Abstract – A series of organoindium-phosphides including $(Me_3CCH_2)_2InPRR'$ (R = R′ = Et, C_6H_{11}; R = C_6H_{11}, R′ = H; R = Me, R′ = Ph) and $(Me_3SiCH_2)_2InP(Me)Ph$ have been prepared and characterized. The characterization data include partial elemental analyses (C, H), ^1H and ^{31}P NMR and IR spectral studies, physical properties, cryoscopic molecular weight studies (with the exception of $(Me_3CCH_2)_2InP(C_6H_{11})_2$), and X-ray structural studies of $[(Me_3CCH_2)_2InPEt_2]_2$ and $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$. The compound $(Me_3CCH_2)_2InPEt_2$ is observed to be a dimer in benzene solution; $(Me_3CCH_2)_2InP(H)(C_6H_{11})$ exists as a dimer-trimer equilibrium mixture whereas

$(Me_3CCH_2)_2InP(Me)(Ph)$ and $(Me_3SiCH_2)_2InP(Me)(Ph)$ are trimers. NMR studies are consistent with $(Me_3CCH_2)_2InP(C_6H_{11})_2$ being a dimer in benzene solution but the concentration was too low to confirm the conclusion by cryoscopic molecular weight studies. The compound $[(Me_3CCH_2)_2InPEt_2]_2$ crystallizes in the monoclinic space group C2/c with a = 12.972(2)Å, b = 15.539(5)Å, c = 18.255(3)Å, β = 99.84(1)°, V = 3625.6 Å^3 and Z = 4 (dimeric molecules). The molecule contains a planar In_2P_2 core with In-P distances of 2.623(2) and 2.641(2)Å. The compound $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$ crystallizes in the triclinic space group $P\bar{1}$ with a = 11.272(3)Å, b = 13.992(5)Å, c = 20.539(9)Å, α = 95.37(5)°, β = 104.83(4)°, γ = 109.14(4)°, V = 2901.9 Å^3 and Z = 2 (trimeric molecules). The molecule has an In_3P_3 six-membered ring which is in the twist-boat conformation with In-P distances ranging from 2.613(3) to 2.659(2)Å.

Group 13-15 compounds of the type $R_2MER'_2$ have been used as single-source precursors[1-7] for ceramic and/or electronic materials and as amphoteric ligands for main-group elements compounds[8-16] and for transition metals.[17,18] In order for these types of compounds to be used most effectively for either of these purposes, it is necessary to first understand the advantages, disadvantages and limitations of the available preparative routes. Secondly, as these compounds are typically associated as dimers but occassionally trimers, the nature of their association and the thermodynamic factors which influence the different degrees of association and their stabilities in the different states of matter must be defined and fully understood. The monomer should be the more useful species for the preparation of materials by chemical vapor deposition because it should be more volatile. Furthermore, it is only the monomer which can be nominally classified as an amphoteric ligand. Since very few monomeric compounds[19,20] of the type $R_2MER'_2$ have been observed, experimental observations of compounds which identify the existence of different degrees of association under different conditions or which identify equilibria between species of different degrees of association under a given set of conditions might be important for using these compounds for the above purposes.

A series of organoindium-phosphides, some of which are substituted with bulky ligands, $(Me_3CCH_2)_2InPEt_2$, $(Me_3CCH_2)_2InP(C_6H_{11})_2$, $(Me_3CCH_2)_2InP(H)(C_6H_{11})$, $(Me_3CCH_2)_2InP(Me)(Ph)$ and $(Me_3SiCH_2)_2InP(Me)(Ph)$ have been prepared in nearly quantitative yields by elimination reactions between the appropriate indium derivative (InR_3) and the phosphine. All reactions were carried out in heated benzene solutions such that the eliminated hydrocarbon (CMe_4 or $SiMe_4$) could be isolated, identified and weighed. The less bulky phosphines, $HPEt_2$ and $H_2P(C_6H)_{11}$ were heated with $In(CH_2CMe_3)_3$ at

50 °C for 40 h each to achieve 96% yields of the corresponding products whereas HP(Me)(Ph) had to be heated with either $In(CH_2CMe_3)_3$ or $In(CH_2SiMe_3)_3$ at 80 °C for 4 days to achieve better than 95% yields of products. In contrast, when the most bulky phosphine, $HP(C_6H_{11})_2$ was allowed to react with $In(CH_2CMe_3)_3$, heating for 21 days at 60 °C was required to achieve a yield of higher than 90%. Heating for only 7 days provided a 43% yield whereas an additional 7 days increased the yield to only 78%. Thus, the elimination reaction between the organoindium compound and the phosphine appears to be the best reaction for preparing the indium phosphide products of highest purity for potential utilization as precursors for the chemical vapor deposition of electronic materials. This preparative method should not introduce any impurity which was not previously present in the indium or phosphorus precursors, the simplest reagents for the OMCVD process.

All compounds prepared in this investigation were as fully characterized as possible. X-ray structural studies of $[(Me_3CCH_2)_2InPEt_2]_2$ and $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$, cryoscopic molecular weight studies in benzene solution and ^{31}P and ^{1}H NMR spectral studies including some variable temperature studies were used to define the degrees of association of the compounds in the solid state and in solution, as appropriate. Physical properties, partial elemental analyses (C and H) and infrared spectra were used to define the identity and purity of the products.

Crystals of dineopentyl indium diethylphosphide are composed of discrete dimeric units of the formula $[(Me_3CCH_2)_2InPEt_2]_2$. The labelling of the atoms in the molecule are shown in Figure 1 and the interatomic bond distances and angles are listed in Tables 1 and 2. There are no abnormally close contacts in the unit cell. The In-P bond distances, 2.623(2) and 2.641(2)Å, are compared with the In-P distances in a variety of other dimers

and trimers in Table 3. The four-membered ring is planar as are all other $(R_2InPR'_2)_2$ compounds which have been structurally characterized (Table 3). The angles within the ring are smaller than the regular tetrahedral angle with observed values of In-P(1)-In(a) = 97.51(9)°, In-P(2)-In(a) = 96.66(8)° and P(1)-In-P(2) = 82.91(6)°.

Cryoscopic molecular weight studies and NMR spectral data indicate that $(Me_3CCH_2)_2InPEt_2$ exists as dimeric species in benzene solution as was observed in the solid state. The molecular weight and NMR (^1H and ^{31}P) spectral data were independent of concentration, an observation which suggests the absence of species other than dimer. The ^1H spectrum had lines for the methyl and methylene protons on phosphorus which were a triplet of doublets of doublets and a quartet of doublets of doublets, respectively. Thus, the lines for methyl group appeared as an pentet centered at 1.02 ppm with a coupling constant of 7.4 Hz and the methylene lines as an apparent quartet of triplets at 1.79 ppm ($^2J_{HCH}$ = 75. Hz, $^2J_{PCH}$ = 2.0 Hz). The methyl protons of the neopentyl groups was a singlet at 1.22 ppm whereas the methylene protons of the neopentyl groups was a triplet at 1.20 ppm ($^3J_{PGa\text{-}CH}$ = 2.1 Hz). The triplet for the methylene protons of the neopentyl group has been observed previously for other related derivatives[21,27] and arises from coupling between the protons and the two phosphorus atoms. Similar coupling may explain the multiplicities of the lines for the CH_2 and CH_3 protons of the ethyl groups bound to phosphorus. The $^{31}P\{^1H\}$ NMR spectrum in d^6-benzene was a single line at -52.2 ppm.

The monocyclohexylphosphide derivative $(Me_3CCH_2)_2InP(H)(C_6H_{11})$ was isolated as a colorless crystalline solid which sublimed at 80 °C under high vacuum. Upon further heating, the compound underwent a glass transition at 120-125 °C and then melted over a

relatively broad range of 131-140 °C. Similar phase changes or glass transitions have been observed for other group 13-15 compounds[28-31] including some neopentyl derivatives.[22,27] A glass transition, however, was not observed for $[(Me_3CCH_2)_2InPEt_2]_2$. These glass transitions have been suggested to be due to changes in the degree of association of the compound with heating.[29,30] After $(Me_3CCH_2)_2InP(H)(C_6H_{11})$ melted, further heating caused the sample to turn yellow with bubbling at 150-170 °C. Then, the sample resolidified. These observations suggest the elimination of additional neopentane but further investigations of this reaction were not pursued.

Crystals of dineopentylindium monocyclohexylphosphide consisted of discrete trimeric units of the formula $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$, which are mutually separated by normal Van der Waals distances. The labelling of the atoms in the molecule are shown in Figure 2 whereas interatomic bond distances and angles are listed in Tables 4 and 5. The In_3P_3 ring for $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$ has a twist boat conformation. The internal ring angles are distorted from the regular tetrahedral angle and range from 128.90(9)-132.18(8)° for In-P-In and 92.85(8)-101.18(9)° for P-In-P, respectively. The ring conformation shown in Figure 2 reveals that the cyclohexyl substituents occupy the E,E,A (equatorial, equatorial, axial) positions on the In_3P_3 ring (twist boat). Similar observations of E,E,A positions for the cyclohexyl group in $[(Me_3Si)_2AlP(H)(C_6H_{11})]_3$[32] have been reported also. It has been suggested that the cyclohexyl groups in the E,E,A positions on the twist boat conformation minimize the steric interactions between the cyclohexyl groups and the substituents on the metal.[32] Other angles including C-In-C (range from 125.9(5)-129.9(5)°) and In-C-C (range from 119.4(6)-125(1)°) are also distorted from the ideal tetrahedral value of 109.47°.

Comparisons between the In-P bond lengths in $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$ and indium phosphide dimers (Table 3) suggest that no correlation exists between In-P bond distances and the degree of association. The In-P bond distances for $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$ range from 2.613(3) to 2.659(2)Å. These In-P distances are comparable to the In-P bond distances in $[(Me_3CCH_2)_2InPEt_2]_2$ of 2.623(2)Å and 2.641(2)Å and all the other indium phosphide dimers. However, the In-P bond distances for $[(Me_3CCH_2)_2InPPh_2]_3$[21] of 2.677(1)Å and 2.699(2)Å are longer.

Concentration dependent ^{31}P NMR spectra and molecular weight studies suggest that $(Me_3CCH_2)_2InP(H)(C_6H_{11})$ exists as a dimer-trimer equilibrium in benzene solution. Two sets of lines were observed in the $^{31}P\{^1H\}$ NMR spectrum at concentrations of 0.043 to 0.151 m. The $^{31}P\{^1H\}$ NMR spectrum of the 0.043 m solution exhibited two lines at -82.6 and -90.7 ppm assigned to phosphorus atoms of the *cis* and *trans* isomers of the dimer and eight lines from -112.3 to -114.4 ppm assigned to the phosphorus atoms in the multiple conformations of the ring and/or orientations of substituents on the trimer. The ratio of the peak heights for the dimer to trimer lines was 1.0:5.3, respectively. When the concentration was increased to 0.151 m, the spectrum revealed similar lines but the ratio of the peak heights for the dimer to trimer decreased (1.0:14.8) but the chemical shifts of the lines did not change. The $^{31}P\{^1H\}$ NMR spectrum of the 0.043 m solution was also investigated as a function of temperature, 20° to 60°. As the temperature increased, the ratio of peak heights of the dimer to trimer increased. This observation suggests the dimer to be more stable relative to the trimer at the higher temperature. Thus, enthalpies of solvation and entropies should be important factors. Since the ^1H NMR spectrum revealed multiple lines for both

the neopentyl and cyclohexyl groups which overlapped, assignments of the resonances to specific protons was not attempted.

The compounds $(Me_3CCH_2)_2InP(Me)Ph$ and $(Me_3SiCH_2)_2InP(Me)Ph$ were prepared readily by elimination reactions between HP(Me)Ph and $In(CH_2CMe_3)_3$ or $In(CH_2SiMe_3)_3$, respectively. Dineopentylindium methylphenylphosphide was a colorless glassy solid which melted to a viscous oil at 30-52 °C whereas $(Me_3SiCH_2)_2InP(Me)Ph$ was a colorless viscous oil at room temperature. Cryoscopic molecular weight studies suggest that both $(Me_3CCH_2)_2InP(Me)Ph$ and $(Me_3SiCH_2)_2InP(Me)Ph$ are trimeric in benzene solution. The complexity of the ^1H and ^{31}P NMR spectra of $(Me_3CCH_2)_2InP(Me)Ph$ and $(Me_3SiCH_2)_2InP(Me)Ph$ indicate a mixture of isomers of trimeric species in benzene. These isomers arise from the different orientations of the methyl and phenyl groups and/or different conformations of the ring.

The elimination reaction has also been utilized for the preparation of $(Me_3CCH_2)_2InP(C_6H_{11})_2$ from $In(CH_2CMe_3)_3$ and $HP(C_6H_{11})_2$ but heating in benzene at 60 °C for 21 days was required. The product was isolated in high yield (ca. 89%) as a colorless crystalline solid that was insoluble at room temperature in typical solvents including hydrocarbons, THF, Et_2O, and aromatic solvents. These solubility characteristics precluded cryoscopic molecular weight studies and extensive NMR studies. The crystalline solid had elemental analyses consistent with the empirical formula.

The limited experimental observations suggest that $(Me_3CCH_2)_2InP(C_6H_{11})_2$ exists as a dimer. The $^{31}P\{^1H\}$ NMR spectrum of $(Me_3CCH_2)_2InP(C_6H_{11})_2$ in d^8-toluene at 70 °C revealed a singlet at -13.00 ppm. Since the chemical shift for $(Me_3CCH_2)_2InP(C_6H_{11})_2$ was only 2.43 ppm down field from the corresponding gallium analog[33]

$[(Me_3CCH_2)_2GaP(C_6H_{11})_2]_2$, which was dimeric in benzene solution, $(Me_3CCH_2)_2InP(C_6H_{11})_2$ might be expected to be dimeric also. The weak Lewis basicity and large size of dicyclohexylphosphide would be expected to favor a low degree of association. It should be noted that $(Me_3CCH_2)_2InP(C_6H_{11})_2$ does not melt below 200 °C but decomposes at 225-229 °C. These observations suggest that $(Me_3CCH_2)_2InP(C_6H_{11})_2$ is not a simple monomer. It is noteworthy that $[Me_2InP(t\text{-}Bu)_2]_2$ exists as a dimer in the solid state and it is also insoluble in Et_2O, hydrocarbons and aromatic solvents.[23]

EXPERIMENTAL

All compounds described in this investigation were extremely sensitive to oxygen and moisture and were manipulated in a standard vacuum line or under a purified argon atmosphere. The compounds $In(CH_2SiMe_3)_3$[34] and $In(CH_2CMe_3)_3$[35] were prepared and purified by literature methods. Dicyclohexylphosphine was purchased from Alfa Products; HP(Me)Ph was purchased from Strem Chemicals, Inc., and $HPEt_2$ and $H_2P(C_6H_{11})$ were generous gifts from the Eastman Kodak Company. All phosphines were purified by distillation prior to use. Solvents were dried by conventional procedures. Elemental analyses were performed by E+R Microanalytical Laboratory, Inc. Corona, NY or Schwarzkopf Microanalytical Laboratory, Woodside, New York. Infrared spectra of Nujol mulls between CsI plates were recorded by means of a Perkin-Elmer 683 spectrometer. The ^1H spectra was recorded at 300 MHz by using a Varian Gemini-300 spectrometer. Proton chemical shifts are reported in δ units (ppm) and are referenced to $SiMe_4$ at δ 0.00 ppm and C_6D_6 at δ 7.15 ppm. The ^{31}P NMR spectrum was recorded at 161.9 MHz on a Varian VXR-400 spectrometer. Proton-decoupled ^{31}P NMR spectra were referenced to 85%

H_3PO_4 at δ 0.00 ppm. All samples for NMR spectra were contained in sealed NMR tubes. Melting points were observed in sealed capillaries. Molecular weights were measured cryoscopically in benzene by using an instrument similar to that described by Shriver and Drezdzon.[36]

Synthesis of $(Me_3CCH_2)_2InPEt_2$. A tube with a Teflon valve was charged with 0.641 g (7.11 mmol) of $HPEt_2$. A 100-mL Schlenk flask equipped with a 20 mm Solv-Seal joint was charged with 2.335 g (7.111 mmol) of $In(CH_2CMe_3)_3$ in the glove box. The Schlenk flask was capped, cooled to -196 °C and evacuated. The $HPEt_2$ was vacuum distilled into the Schlenk flask along with 30 mL of benzene. The reaction mixture was warmed to ambient temperature, and then the Schlenk flask was immersed into a 50 °C oil bath for 6 days. The volatile components were removed and fractionated through two -78 °C traps into a -196 °C trap. Neopentane (0.466 g, 6.46 mmol, 90.9% yield based on $In(CH_2CMe_3)_3$) was isolated in the -196 °C trap and identified by ^1H NMR spectroscopy. The Schlenk flask was fitted with a medium frit equipped with a Schlenk receiving vessel. One extraction with 30 mL of benzene provided a soluble, colorless solid $(Me_3CCH_2)_2InPEt_2$ (2.080 g, 6.907 mmol, 97.13% yield based on $In(CH_2CMe_3)_3$). Crystallographic quality crystals were obtained from a saturated pentane solution maintained at -10 °C. **$(Me_3CCH_2)_2InPEt_2$.** mp 105-106 °C, sublimes at 70 °C, 0.01 mm. ^1H NMR (C_6D_6, δ): 1.02 (tdd, see Discussion, $^2J_{HCH}$ = 7.4 Hz, 2.7 H, $-CH_3$), 1.20 (t, $^3J_{PGaCH}$ = 2.1 Hz, 2.0 H, In-CH_2-), 1.22 (s, 7.0 H, $-CMe_3$), 1.79 (qdd, see Discussion, $^2J_{HCH}$ = 7.5 Hz, $^2J_{PCH}$ = 2.0 Hz, 1.7 H, P-CH_2-). $^{31}P\{^1H\}$ NMR (C_6D_6, δ): -52.19 (s). Anal. Calcd.: C, 48.45; H, 9.24. Found: C, 48.44; H, 9.37. Cryoscopic molecular weight, formula weight 346.52 (obsd molality, obsd mol wt, association): 0.0805, 746, 2.15; 0.0658, 744, 2.15; 0.0529, 745, 2.15. IR (Nujol mull, cm^{-1}):

2725 (vw), 2698 (vw), 1412 (m), 1352 (vs), 1210 (sh), 1105 (m), 1092 (m), 1036 (s), 1020 (m), 1008 (m), 968 (m), 926 (w), 907 (w), 757 (m), 739 (m), 717 (m), 689 (vs), 680 (vs), 660 (m), 568 (m), 555 (sh), 448 (w), 258 (vw), 237 (w).

Synthesis of $(Me_3CCH_2)_2InP(H)(C_6H_{11})$. The compound $(Me_3CCH_2)_2InP(H)(C_6H_{11})$ was prepared from $In(CH_2CMe_3)_3$ (1.941 g, 5.912 mmol) and $H_2PC_6H_{11}$ (0.687 g, 5.91 mmol) in benzene by using the method previously described for $(Me_3CCH_2)_2InPEt_2$. After the reaction mixture was maintained at 50 °C for 40 h, neopentane CMe_4 (0.409 g, 5.67 mmol, 95.9% yield based on $In(CH_2CMe_3)_3$) was collected. Two extractions with 30 mL of benzene yielded a colorless solid $(Me_3CCH_2)_2InP(H)(C_6H_{11})$ (1.919 g, 5.156 mmol, 90.9% yield based on $In(CH_2CMe_3)_3$). Crystallographic quality crystals were obtained from a saturated benzene solution that was prepared by heating the same sample to 50 °C and then cooling the solution to ambient temperature. **$(Me_3CCH_2)_2InP(H)(C_6H_{11})$.** mp glass transition 120-125 °C, melts at 131-140 °C, bubbles at 150-170 °C. ^1H NMR (C_6D_6, δ): 1.18 (m, $-CH_2-$), 1.23 (s, $-CMe_3$), 1.28 (s, $-CMe_3$), 1.41 (s, C_6H_{11}), 1.47 (s, C_6H_{11}), 1.51 (d, $^2J_{HCH}$ = 10.5 Hz, C_6H_{11}), 1.67 (s, C_6H_{11}), 2.07 (d, $^2J_{HCH}$ = 10.5 Hz, C_6H_{11}), 2.21 (m, C_6H_{11}), 2.48 (m, P-H), 3.32 (m, P-H). $^{31}P\{^1H\}$ NMR (0.043 m, C_6D_6, δ): -82.62 (s, 2.9), -90.68 (s, 1.7), -106.2 (s, 0.5), -106.7 (s, 0.4), -112.3 (s, 1.0), -112.7 (s, 1.0), -112.9 (s, 1.6), -113.2 (s, 2.3), -113.3 (s, 5.5), -113.8 (s, 8.0), -114.2 (s, 2.8), -114.4 (s, 2.3). $^{31}P\{^1H\}$ NMR (0.043 m at 40 °C, C_6D_6, δ): -82.95 (s, 5.4), -90.90 (s, 3.5), -112.3 (s, 1.0), -112.7 (s, 1.0), -112.9 (s, 1.8), -113.3 (s, 7.1), -113.8 (s, 7.0), -114.2 (s, 2.9), -114.4 (s, 2.4). $^{31}P\{^1H\}$ NMR (0.043 m at 60 °C, C_6D_6, δ): -83.30 (s, 9.1), -91.17 (s, 6.8), -112.2 (s, 1.0), -112.6 (s, 1.2), -112.9 (s, 1.8), -113.3 (s, 10.9), -113.7 (s, 6.8), -114.0 (s, 2.6), -114.3 (s, 1.8). $^{31}P\{^1H\}$ NMR (0.151 m, C_6D_6, δ): -82.3 (s, 1.7), -90.4 (s, 1.0), -106.2 (s, 0.4), -106.7 (s, 0.3), -112.3 (s, 1.1),

-112.8 (s, 1.4), -112.9 (s, 2.2), -113.2 (s, 7.9), -113. 3 (s, 5.7), -114.0 (s, 13.4), -114. 4 (s, 4.4), -114.5 (s, 3.8). Anal. Calcd.: C, 51.62; H, 9.21. Found: C, 51.77; H, 9.22. Cryoscopic molecular weight, formula weight 371.78 (obsd molality, obsd mol wt, association): 0.0754, 1041, 2.80; 0.0584, 1052, 2.83; 0.0441, 1029, 2.76. IR (Nujol mull, cm^{-1}): 2325 (m), 2303 (m), 2290 (m), 1354 (vs), 1292 (m), 1258 (m), 1230 (vs), 1190 (m), 1173 (m), 1105 (s), 1090 (s), 1068 (w), 1045 (w), 1008 (m), 995 (s), 928 (vw), 914 (w), 907 (w), 892 (m), 885 (m), 848 (m), 820 (m), 805 (m), 740 (m), 719 (m), 686 (s), 670 (s), 620 (w), 600 (w), 568 (s), 506 (vw), 445 (m), 375 (w), 343 (m), 290 (m).

Synthesis of $(Me_3CCH_2)_2InP(Me)Ph$. The compound $(Me_3CCH_2)_2InP(Me)Ph$ was prepared from $In(CH_2CMe_3)_3$ (3.321 g, 10.118 mmol) and HP(Me)Ph (1.256 g, 10.118 mmol) in benzene by using the method previously described for $(Me_3CCH_2)_2InPEt_2$. Heating at 80 °C for 4 days gave 0.714 g CMe_4, 9.90 mmol, 97.8% yield based on $In(CH_2CMe_3)_3$. The crude product was extracted with 45 mL of anhydrous pentane to yield $(Me_3CCH_2)_2InP(Me)Ph$ as a colorless material (3.202 g, 8.421 mmol, 83.23% yield based on $In(CH_2CMe_3)_3$). **$(Me_3CCH_2)_2InP(Me)Ph$.** mp: 30-52 °C (169-172 °C dec.). Material was glass-like in appearance and melted to a thick oil over a broad range. ^1H NMR (C_6D_6, δ): 0.95, 1.00, 1.01, 1.05, 1.08 (s, 18 H, $-CMe_3$); 1.20, 1.21, 1.23, 1.26 (4.1 H, $-CH_2-$); 1.72, 1.76 (m, 2.6 H, PMe). $^{31}P\{^1H\}$ NMR (C_6D_6, δ): -97.6 (s, 11.4), -98.1 (s, 1.00), 98.5 (s, 2.19), -98.7 (s, 8.98), -98.9 (s, 5.07), -99.1 (s, 2.05). Anal. Calcd.: C, 53.69; H, 7.97. Found: C, 53.72; H, 8.26. Cryoscopic molecular weight, formula weight 380.26 (obsd molality, obsd mol wt, association): 0.0787, 1099, 2.89; 0.0657, 1065, 2.80. IR (Nujol mull cm^{-1}): 3078 (w), 3058 (w), 1955 (vw), 1938 (vw), 1795 (vw), 1582 (w), 1480 (s), 1431 (s), 1357 (s), 1308 (vw), 1295 (vw), 1270 (vw), 1265 (w), 1233 (m), 1210 (w), 1199 (vw), 1188 (vw), 1152 (vw),

1108 (w), 1095 (w), 1070 (vw), 1025 (w), 1010 (m), 998 (m), 960 (vw), 928 (vw), 908 (vw), 878 (s), 732 (vs), 689 (vs), 675 (m, sh), 665 (m, sh), 612 (vw), 568 (m), 478 (m), 448 (w), 404 (w), 378 (vw), 330 (vw), 310 (vw), 282 (vw).

Synthesis of $(Me_3SiCH_2)_2InP(Me)Ph$. The compound $(Me_3SiCH_2)_2InP(Me)Ph$ was prepared from $In(CH_2SiMe_3)_3$ (0.820 g, 2.18 mmol) and HP(Me)Ph (0.271 g, 2.18 mmol) in benzene by using the method previously described for $(Me_3CCH_2)_2InPEt_2$. Heating at 80 °C for 4 days gave 0.181 g CMe_4, 1.84 mmol, 84.4% yield based on $In(CH_2CMe_3)_3$. The product was extracted with 45 mL of anhydrous pentane to yield $(Me_3SiCH_2)_2InP(Me)Ph$ as a viscous oil (0.892 g, 2.16 mmol, 99.3% yield based on $In(CH_2SiMe_3)_3$).

$(Me_3SiCH_2)_2InP(Me)Ph$. ^1H NMR (C_6D_6, δ): -0.01, 0.03, 0.06, 0.08, 0.10, 0.16, 0.17, 0.18, 0.20, 0.24, 0.26, 0.28, 0.29, 0.37, 0.38 (s, combined lines 22 H, $-CH_2SiMe_3$); 1.81, 1.82, 1.84, 1.87 (s, 3 H, -PMe). $^{31}P\{^1H\}$ NMR (C_6D_6, δ): -69.6 (s, 1.6), -69.9 (s, 3.1), -70.5 (s, 3.0), -70.8 (s, 1.3), -92.2 (s, 7.9), -98.7 (s, 50.7), -99.2 (s, 96.3). Anal. Calcd.: C, 43.68; H, 7.35. Found: C, 43.49; H, 7.43. Cryoscopic molecular weight, formula weight 412.42 (obsd molality, obsd mol wt, association): 0.0702, 1196, 2.90; 0.0562, 1192, 2.89; 0.0438, 1233, 2.99. IR (Nujol mull cm^{-1}): 3072 (w), 3056 (w), 1562 (w), 1480 (m), 1431 (m), 1424 (m, sh), 1349 (w), 1330 (vw), 1308 (vw), 1275 (w, br), 1252 (m), 1240 (vs), 1152 (vw), 1068 (vw), 1022 (vw), 996 (vw), 955 (m, br), 878 (m), 848 (vs), 820 (vs), 745 (m), 731 (s), 718 (m), 688 (m), 678 (m), 605 (vw), 560 (w), 550 (w), 475 (w), 352 (vw), 345 (vw), 338 (vw), 325 (vw).

Synthesis of $(Me_3CCH_2)_2InP(C_6H_{11})_2$. In the glove box, tared screw-cap vials were charged with 0.721 g (3.64 mmol) of $HP(C_6H_{11})_2$ and with 1.194 g (3.636 mmol) of $In(CH_2CMe_3)_3$. The contents of each vial was transferred quantitatively into a 100-mL Schlenk flask equipped with a 20-mm Solv-Seal joint by using 5 separate 3 mL washings of

benzene. The Schlenk flask was capped, cooled to -196 °C, and evacuated. The reaction mixture was heated with a 60 °C oil bath for 7 days. The volatile component were removed and fractionated through two -78 °C traps into a -196 °C trap. Neopentane (0.112 g, 1.56 mmol, 42.9% yield based on $In(CH_2CMe_3)_3$) was isolated in the -196 °C trap. The neopentane and benzene were vacuum distilled back into the original Schlenk reaction flask and heated at 60 °C for another 7 days. Fractionation yielded 0.206 g CMe_4, 3.42 mmol, 78.4% yield based on $In(CH_2CMe_3)_3$). Again, the neopentane and benzene were vacuum distilled into the original flask and heated at 60 °C. After another week (21 days total) fractionation yielded 0.247 g CMe_4, 3.42 mmol, 94.1% yield based on $In(CH_2CMe_3)_3$. The product was washed with 20 mL of anhydrous pentane to leave $(Me_3CCH_2)_2InP(C_6H_{11})_2$ as a colorless crystalline solid (1.466 g (3.227 mmol, 88.76% yield based on $In(CH_2CMe_3)_3$)). **$(Me_3CCH_2)_2InP(C_6H_{11})_2$.** mp 225-229 °C dec. $^{31}P\{^1H\}$ NMR (70 °C, $C_6D_5CD_3$, δ): -13.00 (s). Anal. Calcd.: C, 58.15; H, 9.76. Found: C, 58.41; H, 9.41. IR (Nujol mull, cm^{-1}): 2700 (vw), 1340 (m), 1334 (m), 1327 (m), 1296 (w), 1290 (m), 1257 (m), 1227 (s), 1208 (m), 1186 (m), 1172 (s), 1163 (m), 1094 (m), 1089 (sh), 1065 (w), 1042 (w), 1036 (vw), 1020 (w), 1009 (m), 995 (s), 922 (vw), 910 (w), 901 (w), 880 (m), 844 (s), 813 (vw), 780 (vw), 737 (s), 725 (m), 671 (vs), 640 (s), 507 (w), 448 (m), 430 (sh), 380 (w), 350 (w), 318 (vw), 288 (m). Solubility: trace solubility in aromatic solvents and insoluble in Et_2O, THF and hydrocarbons.

X-ray Data Collection, Structure Determination and Refinement for $[(Me_3CCH_2)_2InPEt_2]_2$. A transparent single crystal of $[(Me_3CCH_2)_2InPEt_2]_2$ was mounted in a thin-walled glass capillary under Ar and transferred to the goniometer. The space group was determined to be either the centric C2/c or acentric Cc from the systematic

absences. The subsequent solution and successful refinement of the structure was carried out in the centric space group C2/c. A summary of data collection parameters is given in Table 6.

Least-squares refinement with isotropic thermal parameters led to R = 0.066. Disorder was obvious in the neopentyl group C(6)-C(10). The disorder resolved itself into two orientations of C(6), C(9), and C(10). C(7) and C(8) were common to both. Refinement of the occupancy factors gave C(6), C(9), C(10) at 55% and their primed counterparts at 45%. The two orientations were refined in alternate least-squares cycles. The hydrogen atoms were not included in the final refinement. Refinement of nonhydrogen atoms with anisotropic temperature factors led to the final values of R = 0.045 and R = 0.076. The final values of the positional parameters are given in Table 7.

X-ray Data Collection, Structure Determination and Refinement for $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_2$. A transparent single crystal of $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_2$ was mounted in a thin-walled glass capillary under Ar and transferred to the goniometer. The space group was determined to be either the centric $P\bar{1}$ or acentric P1. The subsequent solution and successful refinement of the structure was carried out in the centric space group $P\bar{1}$. A summary of data collection parameters is given in Table 8.

High thermal motion was noted for almost all C atoms. The only resolvable disorder was two orientations for the C(31)-C(36) cyclohexyl group. C(34)-C(36) and C(34)'-C(36)' were refined with 50% occupancy in alternate least-squares cycles. Due to the thermal motion and disorder the hydrogen atoms were not included in the final refinement. Refinement of nonhydrogen atoms with anisotropic temperature factors led to the final

values of R = 0.054 and R_w = 0.078. The final values of the positional parameters are given in Table 9.

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Supplementary Material. Listings of anisotropic thermal parameters and bond distances and angles (pages); a listing of observed and calculated structure factors for $[(Me_3CCH_2)_2InPEt_2]$ and $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$.

Table 1. Interatomic Distances (Å) for $[(Me_3CCH_2)_2InPEt_2]_2$.				
(A)	Indium-Phosphorus Distances			
	In-P(1)	2.623(2)	In-P(2)	2.641(2)
(B)	Indium-Carbon Distances			
	In-C(1)	2.211(8)	In-C(6)	2.17(2)
	In-C(6)'[a]	2.29(3)		
(C)	Phosphorus-Carbon Distances			
	P(1)-C(11)	1.869(9)	P(2)-C(13)	1.835(8)

[a]The neopentyl group C(6)-C(10) is disordered.

Table 2. Angles (deg) for $[(Me_3CCH_2)_2InPEt_2]_2$.

(A)	Angles around the Indium Atom			
	P(1)-In-P(2)	82.91(6)	P(1)-In-C(1)	111.1(2)
	P(1)-In-C(6)	115.7(4)	P(1)-In-C(6)′	101.2(7)
	P(2)-In-C(1)	100.5(2)	P(2)-In-C(6)	124.6(4)
	P(2)-In-C(6)′	118.1(6)	C(1)-In-C(6)	116.9(5)
	C(1)-In-C(6)′	132.2(7)		
(B)	Angles around the Phosphorus Atoms			
	In-P(1)-In(a)	97.51(9)	In-P(2)-In(a)	96.66(8)
	In-P(1)-C(11)	113.5(3)	In-P(2)-C(13)	120.4(3)
	In-P(1)-C(11a)	113.5(3)	In-P(2)-C(13a)	107.9(3)
	C(11)-P(1)-C(11a)	105.5(5)	C(13)-P(2)-C(13a)	104.5(5)
(C)	Indium-Carbon-Carbon Angles			
	In-C(1)-C(2)	118.7(6)	In-C(6)-C(7)	123(1)
			In-C(6)′-C(7)	119(1)
(D)	Phosphorus-Carbon-Carbon Angles			
	P(1)-C(11)-C(12)	112.1(7)	P(2)-C(13)-C(14)	113.3(6)

[a]Symmetry code: -x, y, 1/2-z.

Table 3. Indium-Phosphorus Bond Distances for Compounds of the Type $[R_2InPR'_2]_n$.

Compound	d InP(Å)		Reference
$[(Me_3CCH_2)_2InPEt_2]_2$	2.623(2) 2.641(2)		this work
$[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$	2.613(3) 2.625(3) 2.644(3)	2.637(3) 2.645(3) 2.659(2)	this work
$[(Me_3CCH_2)_2InPPh_2]_3$	2.677(1) 2.699(2)		21
$[(Me_3SiCH_2)_2InPPh_2]_2$	2.664(2) 2.643(2)	2.659(2) 2.632(2)	22
$[Me_2InP(t\text{-}Bu)_2]_2$	2.656(4) 2.637(4)		23
$[Et_2InP(t\text{-}Bu)_2]_2$	2.635(2)		24
$[(Me_3SiCH_2)_2InP(SiMe_3)_2]_2$	2.654(2) 2.656(2)		13
$[(Me_3SiCH_2)_2InP(H)(t\text{-}Bu)]_2$	2.633(1) 2.638(1)		25
$[(C_5Me_5)(Cl)InP(SiMe_3)_2]_2$	2.648(2) 2.594(1)		26

Table 4. Interatomic Distances (Å) for $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$.

(A)	Indium-Phosphorus Distances			
	In(1)-P(1)	2.644(3)	In(1)-P(3)	2.613(3)
	In(2)-P(2)	2.645(3)	In(2)-P(1)	2.637(3)
	In(3)-P(3)	2.659(2)	In(3)-P(2)	2.625(3)
(B)	Indium-Carbon Distances			
	In(1)-C(1)	2.195(8)	In(1)-C(6)	2.22(1)
	In(2)-C(11)	2.20(1)	In(2)-C(16)	2.19(2)
	In(3)-C(21)	2.188(9)	In(3)-C(26)	2.16(1)
(C)	Phosphorus-Carbon Distances			
	P(1)-C(31)	1.83(2)	P(2)-C(37)	1.88(1)
	P(3)-C(43)	1.871(7)		

Table 5. Angles (deg) for $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$.

(A)	Angles around the Indium Atoms			
	P(1)-In(1)-P(3)	101.18(9)	P(1)-In(1)-C(1)	98.1(3)
	P(3)-In(1)-C(1)	106.5(3)	P(1)-In(1)-C(6)	104.4(3)
	P(3)-In(1)-C(6)	113.3(3)	C(1)-In(1)-C(6)	128.6(5)
	P(1)-In(2)-P(2)	92.85(8)	P(1)-In(2)-C(11)	105.5(3)
	P(2)-In(2)-C(11)	104.5(3)	P(1)-In(2)-C(16)	118.3(4)
	P(2)-In(2)-C(16)	103.5(5)	C(11)-In(2)-C(16)	125.9(5)
	P(2)-In(3)-P(3)	93.69(8)	P(2)-In(3)-C(21)	98.4(3)
	P(3)-In(3)-C(21)	108.0(3)	P(2)-In(3)-C(26)	113.9(3)
	P(3)-In(3)-C(26)	107.0(3)	C(21)-In(3)-C(26)	129.9(5)
(B)	Angles around the Phosphorus Atoms			
	In(1)-P(1)-In(2)	130.1(1)	In(2)-P(1)-C(31)	107.2(5)
	In(2)-P(2)-In(3)	132.18(8)	In(1)-P(1)-C(31)	107.6(6)
	In(2)-P(2)-C(37)	112.1(4)	In(3)-P(2)-C(37)	107.0(4)
	In(1)-P(3)-In(3)	128.90(9)	In(1)-P(3)-C(43)	105.8(3)
	In(3)-P(3)-C(43)	115.1(3)		
(C)	Indium-Carbon-Carbon Angles			
	In(1)-C(1)-C(2)	120.4(6)	In(1)-C(6)-C(7)	120.5(8)
	In(2)-C(11)-C(12)	119.7(7)	In(2)-C(16)-C(17)	125(1)
	In(3)-C(21)-C(22)	121.3(6)	In(3)-C(26)-C(27)	119.4(6)
(D)	Phosphorus-Carbon-Carbon Angles			
	P(1)-C(31)-C(32)	114(1)	P(1)-C(31)-C(36)	122(1)
	P(1)-C(31)-C(36)′	117(1)	P(2)-C(37)-C(38)	112.4(8)
	P(2)-C(37)-C(42)	113(1)	P(3)-C(43)-C(44)	110.6(6)
	P(3)-C(43)-C(48)	111.6(6)		

Table 6. Crystal Data and Summary of Intensity Data Collection and Structure Refinement of $[(Me_3CCH_2)_2InPEt_2]_2$.

molecular formula	$C_{28}H_{64}In_2P_2$
color/shape	colorless/parallelepiped
mol wt	684.34
space group	C2/c
temp., °C (K)	22 (295)
cell constants[a]	
a, Å	12.972(2)
b, Å	15.539(5)
c, Å	18.255(3)
β, deg	99.84(1)
V, Å^3	3625.6
Z	4
D_{calc}, g cm^{-3}	1.25
μ_{calc}, cm^{-1}	13.6
diffractometer/scan	Enraf-Nonius CAD-4/ω-2θ
range of relative transm factors, %	89/100
radiation, graphite monochromator (λ, Å)	MoKα (λ = 0.71073)
max cryst dimens, mm	0.20 × 0.30 × 0.35
scan width	0.80 + 0.35tanθ
std reflcns	600; 080; 0, 0, 14
stds measd	±2%
no. of reflcns measd	3454
2θ range, deg	2 ≤ 2θ ≤ 50
range of h, k, l	+15, +18, ±21 (h+k = 2n only)
no. of reflcns observed [$F_o \geq 5\sigma(F_o)$][b]	2261
computer programs[c]	SHELX[37]
structure solution	SHELXS[38]
no. of params varied	175
weights	$[\sigma(F_o)^2 + 0.002\ F_o^2]^{-1}$
GOF	1.10
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.045
R_w	0.076
largest feature in final diff map, eÅ^{-3}	0.6

[a]Least-squares refinement of $((\sin\theta)/\lambda)^2$ values for 25 reflections $\theta > 19°$.
[b]Corrections: Lorentz-polarization and absorption (empirical, ψ scan).
[c]Neutral scattering factors and anomalous dispersion corrections from ref 39.

Table 7. Final Fractional Coordinates for $[(Me_3CCH_2)_2InPEt_2]_2$.

Atom	x/a	y/b	z/c	B(eqv)[a]
In	0.15096(4)	0.87403(3)	0.24587(3)	2.47
P(1)	0.0000	0.7627(2)	0.2500	2.65
P(2)	0.0000	0.9870(2)	0.2500	2.25
C(1)	0.2453(7)	0.8929(6)	0.3576(5)	3.66
C(2)	0.3422(6)	0.8353(5)	0.3815(4)	2.68
C(3)	0.393(1)	0.8615(9)	0.4616(7)	6.01
C(4)	0.4219(8)	0.8468(8)	0.3304(6)	4.78
C(5)	0.3114(9)	0.7398(6)	0.3801(7)	5.26
C(6)	0.236(1)	0.860(1)	0.1540(8)	3.33
C(6)′[b]	0.196(2)	0.843(2)	0.133(1)	5.24
C(7)	0.2110(8)	0.9139(7)	0.0823(5)	4.35
C(8)	0.2780(9)	0.8895(7)	0.0234(6)	5.09
C(9)	0.093(1)	0.889(2)	0.050(1)	5.21
C(10)	0.208(2)	1.004(1)	0.091(1)	6.26
C(9)′	0.125(2)	0.956(2)	0.048(1)	7.72
C(10)′	0.293(2)	0.988(2)	0.136(1)	7.15
C(11)	0.0242(7)	0.6899(5)	0.3327(5)	3.59
C(12)	-0.070(1)	0.6320(6)	0.3376(8)	5.40
C(13)	-0.0419(6)	1.0592(5)	0.1710(4)	3.10
C(14)	-0.1284(9)	1.1242(6)	0.1846(6)	4.56

[a] $B(eqv) = 4/3[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}]$

[b] Primed and unprimed atoms of like numbers are disordered. C(6), C(9), and C(10) are present at 55% occupancy, while their primed counterparts have 45% occupancy.

Table 8. Crystal Data and Summary of Intensity Data Collection and Structure Refinement of $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$.

molecular formula	$C_{48}H_{69}In_3P_3$
color/shape	colorless/parallelepiped
mol wt	1116.7
space group	$P\bar{1}$
temp., °C (K)	20 (293)
cell constants[a]	
a, Å	11.272(3)
b, Å	13.992(5)
c, Å	20.539(9)
α, deg	95.37(5)
β, deg	104.83(4)
γ, deg	109.14(4)
V, Å^3	2901.9
Z	2
D_{calc}, g cm^{-3}	1.28
μ_{calc}, cm^{-1}	12.87
diffractometer/scan	Enraf-Nonius CAD-4/ω-2θ
range of relative transm factors, %	85/100
radiation, graphite monochromator (λ, Å)	MoKα (λ = 0.71073)
max cryst dimens, mm	0.15 × 0.30 × 0.35
scan width	0.80 + 0.35tanθ
std reflcns	400; 030; 006
decay of stds	±3%
no. of reflcns measd	10,246
2θ range, deg	$2 \le 2\theta \le 50$
range of h, k, l	+9, +12, ±17
no. of reflcns observed [$F_o \ge 5\sigma(F_o)$][b]	6051
computer programs[c]	SHELX[37]
structure solution	SHELXS[38]
no. of params varied	523
weights	$[\sigma(F_o)^2 + 0.008\ F_o^2]^{-1}$
GOF	0.40
$R = \Sigma\|\|F_o\|-\|F_c\|\|/\Sigma\|F_o\|$	0.054
R_w	0.078
largest feature in final diff map, eÅ^{-3}	1.2 near In(2)

[a]Least-squares refinement of $((\sin\theta)/\lambda)^2$ values for 25 reflections $\theta > 20°$.
[b]Corrections: Lorentz-polarization and absorption (empirical, ψ scan).
[c]Neutral scattering factors and anomalous dispersion corrections from ref 39.

Table 9. Final Fractional Coordinates for $[(CH_3CH_2)_2InP(H)(C_6H_{11})_3$.

Atom	x/a	y/b	z/c	B(eqv)[a]
In(1)	0.11661(6)	0.51323(4)	0.24386(3)	2.77
In(2)	0.15441(6)	0.83677(5)	0.17487(3)	3.27
In(3)	-0.01876(6)	0.74770(4)	0.36161(3)	3.21
P(1)	0.1225(2)	0.6398(2)	0.1559(1)	3.40
P(2)	0.1579(2)	0.8441(2)	0.3045(1)	3.39
P(3)	-0.0307(2)	0.5618(2)	0.3106(1)	3.02
C(1)	-0.010(1)	0.3687(7)	0.1708(5)	3.93
C(2)	0.0029(9)	0.2664(7)	0.1849(5)	3.54
C(3)	0.138(1)	0.269(1)	0.1906(7)	6.03
C(4)	-0.037(1)	0.2435(9)	0.2503(6)	5.98
C(5)	-0.103(1)	0.1781(9)	0.1218(7)	5.75
C(6)	0.326(1)	0.5599(9)	0.3072(5)	4.55
C(7)	0.3606(9)	0.5631(7)	0.3839(5)	3.56
C(8)	0.329(1)	0.648(1)	0.4187(7)	7.11
C(9)	0.511(1)	0.583(1)	0.4120(7)	5.82
C(10)	0.282(2)	0.463(1)	0.4006(8)	7.60
C(11)	0.3602(9)	0.9202(8)	0.1804(6)	4.32
C(12)	0.392(1)	1.0013(8)	0.1354(6)	4.56
C(13)	0.332(2)	0.951(1)	0.0582(7)	6.58
C(14)	0.341(1)	1.0861(9)	0.1515(8)	6.19
C(15)	0.545(1)	1.049(1)	0.156(1)	8.61
C(16)	-0.014(1)	0.880(1)	0.1282(8)	7.44
C(17)	-0.137(1)	0.8091(9)	0.0746(6)	4.80
C(18)	-0.240(1)	0.858(1)	0.0584(9)	8.11
C(19)	-0.117(2)	0.750(2)	0.0157(8)	8.78
C(20)	-0.200(1)	0.720(1)	0.112(1)	8.67
C(21)	0.108(1)	0.7916(7)	0.4684(5)	4.08

Table 9 (continued)

C(22)	0.059(1)	0.7421(7)	0.5257(5)	4.35
C(23)	-0.051(1)	0.775(1)	0.5379(7)	6.94
C(24)	0.019(2)	0.6281(9)	0.5103(7)	7.10
C(25)	0.180(2)	0.784(1)	0.5945(6)	6.61
C(26)	-0.211(1)	0.7545(7)	0.3177(6)	4.57
C(27)	-0.232(1)	0.8600(8)	0.3306(6)	4.87
C(28)	-0.204(2)	0.897(1)	0.4052(8)	8.20
C(29)	-0.147(2)	0.937(1)	0.299(1)	9.11
C(30)	-0.381(2)	0.837(1)	0.292(1)	9.50
C(31)	0.234(2)	0.624(1)	0.1080(6)	8.11
C(32)	0.210(1)	0.657(1)	0.0396(6)	7.38
C(33)	0.264(2)	0.596(2)	-0.010(1)	6.18
C(34)	0.395(3)	0.626(3)	0.027(2)	8.22
C(35)	0.375(2)	0.534(2)	0.0716(9)	4.46
C(36)	0.272(3)	0.535(2)	0.107(1)	5.81
C(33)′[b]	0.332(3)	0.668(2)	0.007(2)	9.60
C(34)′	0.360(3)	0.560(2)	0.016(2)	7.76
C(35)′	0.420(2)	0.580(2)	0.096(1)	6.48
C(36)′	0.344(2)	0.621(2)	0.1433(9)	5.77
C(37)	0.248(1)	0.9778(8)	0.3571(6)	5.01
C(38)	0.224(1)	1.0568(8)	0.3191(7)	5.32
C(39)	0.300(2)	1.167(1)	0.3615(8)	6.58
C(40)	0.435(2)	1.188(1)	0.3982(9)	9.92
C(41)	0.450(1)	1.108(1)	0.4444(7)	6.79
C(42)	0.389(1)	0.997(1)	0.3940(8)	7.59
C(43)	-0.2012(8)	0.4643(6)	0.2694(5)	3.29
C(44)	-0.263(1)	0.4868(7)	0.2018(5)	3.94

Table 9 (continued)

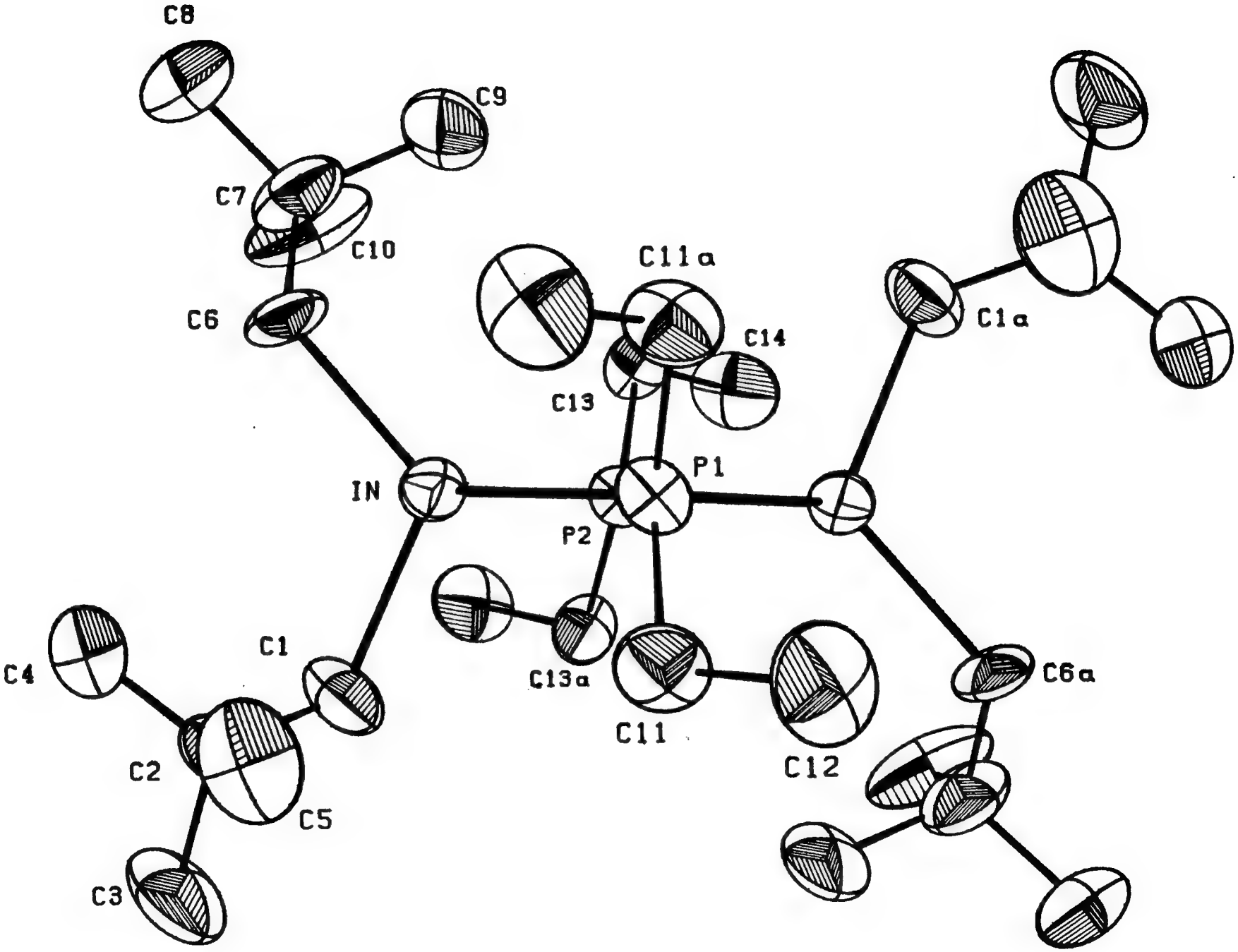
C(45)	-0.400(1)	0.4012(9)	0.1661(6)	4.86
C(46)	-0.489(1)	0.397(1)	0.2118(8)	6.44
C(47)	-0.427(1)	0.3745(9)	0.2797(8)	5.81
C(48)	-0.287(1)	0.4570(7)	0.3175(5)	4.23

[a] $B(eqv) = 4/3[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab(\cos\gamma)\beta_{12} + ac(\cos\beta)\beta_{13} + bc(\cos\alpha)\beta_{23}]$
[b] Primed atoms are disordered with 50% occupancy each.

CAPTIONS FOR FIGURES

Figure 1. Labelling of atoms in $[(Me_3CCH_2)_2InPEt_2]_2$ (ORTEP diagram; 50% ellipsoids).

Figure 2. Labelling of atoms in $[(Me_3CCH_2)_2InP(H)(C_6H_{11})]_3$ (ORTEP diagram; 50% ellipsoids).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C10
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C11a
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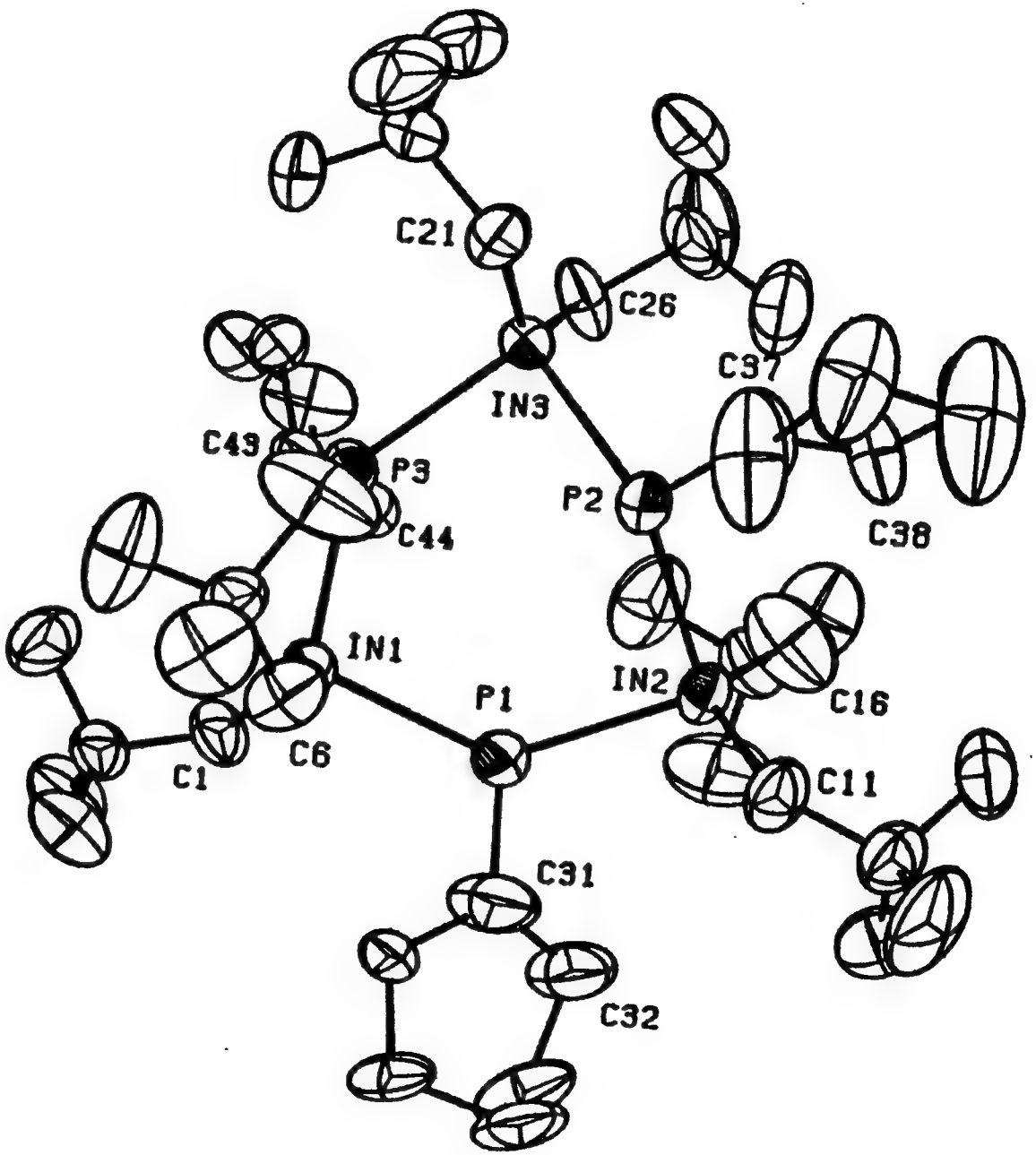
C2
C5
C3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C43
P3
P2
C44
C38
IN1
IN2
C16
P1
C6
C1
C11
C31
C32

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